Factors affecting the water retaining characteristics of lime and cement mortars in the freshly-mixed state

C. Ince[1] (PhD), M. A. Carter[1] (PhD), M. A. Wilson*[1] (PhD), N. C. Collier[2] (PhD), A El-Turki[3] (PhD), R J Ball[3] (PhD) and G C Allen[3] (Prof)

[1] *School of Mechanical, Aerospace and Civil Engineering, The University of Manchester, PO Box 88, Manchester M60 1QD, UK.*

[2] *Department of Engineering Materials, The University of Sheffield, Sheffield S1 3JD.*

[3] *The Interface Analysis Centre, The University of Bristol, Bristol BS2 8BS, UK.*

Phone: +44(0) 161 306 4245

Fax: +44(0) 161 306 4252

Email: moira.wilson@manchester.ac.uk

Abstract

Desorptivity, the parameter which quantifies water retaining ability, has been determined for freshly-mixed natural hydraulic lime and cement mortars using a modified American Petroleum Institute pressure cell. The results show how greatly this ability depends on hydraulicity. Mix composition is also highly significant: smaller proportion of sand, larger grain size of sand and higher proportion of mix water all decrease the water retaining ability. In practice water is abstracted due to the capillary pressure of the masonry unit and the results presented are discussed in terms of typical capillary pressures exerted by clay facing brick. Changes in desorptivity also occur with elapsed time from mixing. The most hydraulic mortars become less water retaining for about 90 minutes after mixing and then, gradually more water retaining.

Keywords: desorptivity; mortar; cement.

* To whom correspondence should be addressed (moira.wilson@manchester.ac.uk)

1 Introduction

Ordinary Portland cement (OPC), the range of natural hydraulic limes (NHL 2, 3.5 and 5) and hydrated lime (CL90) binders may be considered to form a spectrum of related materials. The terminology for building limes is defined by European Standard EN459-1 [1]. The hydraulicity of these binders, or their ability to set under water, is dependent on the amount of clay which is either added during cement manufacture or, in the case of NHLs, present as impurity in the parent limestone. Hydraulicity may be expressed in terms of the CaO: SiO_2 ratio. For the materials used in this study the elemental analysis, determined by XRF,

and their equivalent oxide composition are given in [2]. OPC and NHL mortars set and harden by complex hydration reactions with carbonation (the absorption of CO_2 from the atmosphere) making some contribution to strength gain. Hydrated lime, also known as air lime [1], is not at all hydraulic. Mortars composed of hydrated lime and sand set by loss of water and then harden entirely by carbonation. The hydraulicity of the binder not only affects the properties of a mortar when cured, but also has a strong influence on the water retaining properties of the mortar in its freshly-mixed wet state. We have previously shown [3 - 6] that Portland cement, NHL and hydrated lime mortars of the same water: binder: sand composition by volume have greatly differing water retaining characteristics: OPC mortar, at one end of the spectrum (most hydraulic), is highly water *releasing*; NHL mortars become progressively more water *retaining* as their hydraulicity decreases *i.e.* from NHL 5 to NHL 2; and hydrated lime mortars, at the other end of the spectrum, are strongly water *retaining*.

In practice the extent to which a freshly mixed mortar is dewatered depends not only on its water retaining characteristics but also on the suction of the substrate to which it is applied, either in the form of render or of jointing mortar in masonry. The abstraction of water from the wet mix by the substrate is a critical factor in both early-stage stiffening and mix-substrate adhesion. In previous work [6] we have shown that for OPC and NHL mortars a 10mm mortar joint will be dewatered between 15 and 60 seconds following application to a typical brick substrate and that 40 – 60% of the original mix water will be lost in the process.

We have examined the water retaining characteristics of freshly-mixed mortars using a pressure filtration technique [3, 4] to determine the desorptivity R, which

characterises the water retaining abilities of these materials. High values of R indicate poor water retaining ability, and low values the reverse. The theory of water loss from wet mixes has been developed from analyses of slurry filtration and dewatering used originally in oil field engineering [7 - 11] and the application to construction materials is comprehensively discussed in [12]. In pressure filtration water is forced from the wet mix by an unreactive gas such as nitrogen. Theory [11, 12] predicts that the dewatering of a slurry at constant applied filtration pressure P proceeds as time$^{1/2}$. The cumulative desorbed volume of water per unit area $i = Rt^{1/2}$ where R is the desorptivity. The term i here has the units mm [mm^3/mm^2]. The time, t, has been measured in minutes therefore R has the units mm min$^{-1/2}$. The lower the value of R, the more water retaining the slurry, in this case the mortar. By the end of dewatering, the original wet mix has been replaced by a "filter cake". In the theoretical model we assume that this filter cake is incompressible. The desorptivity varies with the applied gas pressure such that $R = CP^n$, where C is a constant dependent upon the material and the exponent n is a measure of the compressibility of the filter cake with $n = 0.5$ indicating incompressibility [13]. For this value of n, R will show linear dependence on $P^{1/2}$. If the cake is compressible, n will be significantly less than 0.5.

The purpose of this paper is to report a comprehensive investigation of the extent to which the desorptivity is affected by a number of factors: the hydraulicity of the mix; the pressure that is applied to dewater the wet mix; the elapsed time since mixing; and the volumetric mix composition in terms of both binder: sand and water: binder ratios together with the particle size fraction of sand used in the mix. In practical terms these correspond to the nature of the binder, variations in

suction properties of the substrates to which the mortars are applied; the length of time after mixing for which a hydraulic mix remains workable; “maturation” of lime putty; and whether the water retaining ability of a mortar made with a particular binder may be manipulated by changing the proportions and characteristics of the other constituents of the mix.

2 Experimental work

The specific surface areas of ordinary Portland cement (OPC), the full range of natural hydraulic limes (NHL 2, 3.5 and 5) and a hydrated calcium lime (CL 90), all taken from freshly opened containers, were determined from nitrogen sorption by the BET method.

Desorptivity measurements were carried out on freshly mixed mortars of mix proportions 0.78:1:2 water: binder: sand by volume using the full range of binders. The measured bulk density of each binder is given in the second column of Table 1. In these experiments the desorptivity was measured over as wide a range of applied pressures as possible. Additional mixes were prepared in order to investigate variations in desorptivity with changes in sand: binder ratio, water: binder ratio and sand grading. When examining the effect of each of these variables, a single value of applied pressure was selected for the measurement of desorptivity. Full details of the mortar compositions examined are given in the figure captions.

In addition, changes in desorptivity with elapsed time since mixing were measured at a single value of applied pressure. For this study mortars were stored in sealed polythene bags immediately following mixing in order to prevent

evaporation and exclude air as far as possible. Successive desorptivity measurements were carried out on samples taken from these stored mixes until the onset of setting made the mix too stiff to tamp into the pressure cell. For OPC mortars this occurred after about three hours. CL 90 mortars on the other hand, if kept perfectly sealed, should remain unset indefinitely.

Desorptivity measurements were carried out using the modified version of the American Petroleum Institute (API) pressure cell shown in Fig. 1. The length of the cell is 245 mm and its internal diameter 54 mm. The main modification to the original design [14] is the use of threaded collars at the top and bottom which not only allow more rapid assembly and disassembly of the cell but also reduce the possibility of contamination of the O-rings and threads with sand grains. Contamination of this type was a common cause of pressure loss in the original design. As in the original API cell, the outflow end is fitted with a filter paper of particle retention size less than that of the binders under test, wire gauze and drain tap.

2.1 Preparation of the mortars

The mortars were prepared with pre-dried concreting sand. This was a single source (Croxden) sand having 98.9% of particles < 1.18 mm. Mortars were also prepared using the 150 - 300 μm size fraction, referred to in this paper as "sieved sand". In addition a series of NHL 2 mortars was prepared with three other sand particle size fractions. The masses of binder and sand needed to produce the required mix proportions by volume were calculated from the carefully determined values of density given in Table 1. To ensure consistency, a standard mixing regime was followed. The water was placed into the bowl of an orbital paddle mixer and the binder added and mixed for 1 minute. Without stopping the

mixer, sand was then added gradually over the following 1 minute and the resultant mortar mixed for a further minute. The mixer was then stopped and all unmixed solids scraped from the paddle and the sides of the mixing bowl. Mixing was then continued for a further 7 minutes giving a total mixing time of 10 minutes. Between 1.2 and 2 kg of mortar was prepared in each batch, which provided sufficient material for three to five consecutive desorptivity measurements.

2.2 Measurement of desorptivity

The desorptivity of each mortar was measured following the method described in [3]. Briefly, a known volume of freshly mixed mortar was placed into the pressure cell in several layers, each being tamped thoroughly to eliminate voids before the addition of the next. Having added the wet mix, the cell was sealed and pressurized to the required value before opening the drain tap. The desorbed water was collected in a flask on a top loading balance connected to a computer. The mass of the desorbed water was recorded at 10 s intervals until gas-breakthrough occurred. The desorptivity of the mix was determined from the gradient of a graph of the cumulative desorbed volume of water per unit area, i, plotted against the square root of time.

3 Results and discussion

3.1 Hydraulicity and particle size distribution

Table 1 gives the specific surface areas of the Portland cement, NHLs and CL90, from which it can be seen that the latter has a considerably higher value than either cement or hydraulic lime. This implies that particle sizes are much smaller and is supported by particle size distributions supplied by the manufacturer which indicate approximately 90 % of particles < 20 μm for CL90, NHL 2 and NHL 3.5

and approximately 95% < 40 μm for NHL 5 and Portland cement. Based on its small particle size and consequent small radii of curvature of the menisci between particles, CL90 would be expected to have an inherently high water retaining ability.

The measured values of desorptivity for OPC, NHL 5, NHL 2 and CL 90 mortars, prepared with sieved sand are shown in Table 2. These desorptivity measurements were carried out immediately following mixing and at the lower pressure limit of the measurable range in each case. It is not possible to desorb water at all from the CL 90 mortar at applied pressures < 0.2 MPa. At 0.2 MPa the desorptivity of CL90 is approximately 40% of that of OPC mortar despite being dewatered at four times the pressure. It can be seen from Table 2 that OPC mortar (most hydraulic) is less water retaining than equivalent mixes incorporating hydraulic limes (less hydraulic), which in turn are *much* less water retaining than mortar made with hydrated lime. We express these results qualitatively since the high surface area of CL90 may be a contributory factor, but this is the subject of continuing research. These results clearly demonstrate that for a range of mortars of the same composition, the desorptivity immediately after mixing increases dramatically with increasing hydraulicity of binder and fineness of particle size.

3.2 Applied pressure

Fig. 2 shows the variation in desorptivity with applied pressure for CL 90 and NHL 2 mortars of the same mix composition 0.78:1:2 water: binder: sand. (Fig. 2(B) includes the results for a second water: binder ratio, and this is discussed in **3.4.1**). Fig. 2 clearly demonstrates the increase in desorptivity produced by an increase in applied gas pressure. For these non- and weakly- hydraulic binders there is little potential for further increase in desorptivity with increasing pressure

beyond 0.8 MPa for CL90 and 0.25 MPa for NHL 2. (The upper limit of applied pressure for a desorptivity measurement was defined as that at which gas breakthrough occurred within 20 to 30 s, too soon for sufficient data to be collected to determine *R*.) From the equations of the curves of best fit in Fig. 2, the constant C can be seen to increase when there is greater increase in *R* with increasing pressure. The closeness of the exponent to 0.5 in these equations indicates the incompressibility of the filter cake in each case, which may be confirmed if these data are replotted as *R* versus $P^{1/2}$.

In contrast, for the more hydraulic (and more water releasing) NHL 3.5, NHL 5 and OPC mortars shown in Fig. 3 the variation in desorptivity is shown over a smaller range of applied pressure. Desorptivity increased little at pressures above 0.15 MPa for NHL 3.5 mortar or above 0.1 MPa for the other two mortars. In general it can be seen that for freshly mixed mortars desorptivity increases as the applied pressure is increased to some maximum value which is lower for more hydraulic mortars. These particular hydraulic mortars were prepared with sieved sand, hence values of *R* are somewhat lower than those presented in Table 2 (discussed further in **3.4.3**). We are confident from previous work [2] that OPC mortars give incompressible filter cakes. The sharp transition to a plateau region in Fig. 3 is also indicative of incompressibility [13].

3.3 Elapsed time since mixing

Fig. 4 shows the results of successive measurements of the desorptivity of OPC, NHL 5 and NHL 2 mortars, each taken from the same batch, all of the same mix proportions of 0.78:1:2 water: binder: sieved sand over several hours following mixing. After a slight increase over the first hour or so after mixing OPC and NHL 5 mortars showed a progressive decrease in desorptivity with time. The

desorptivity of NHL 2 mortar decreased slightly in three hours but was still measurable twenty four hours after mixing. After this time, although not completely set, the mortar was too stiff to tamp into the pressure cell.

These results show that as mortars stiffen they become more water retaining. Since air is largely excluded, and therefore carbonation prevented, these changes in mortar desorptivity must be due to hydration reactions alone. OPC and NHL 5 mortars, containing higher proportion of hydraulic material than NHL 2 mortar, show a similar decrease in desorptivity with elapsed time since mixing. Desorptivity is thus detecting subtle changes in composition as the mortar stiffens before setting. Reaction products are likely to fill the spaces between particles and thus have some effect on desorptivity. It is also tempting to suggest that we are observing the so called dormant period of cement hydration.

Fig. 5 shows the same data for the CL 90 mortar. It can be seen (inset) that the desorptivity remained almost constant over several hours following mixing. However, measurements at daily intervals showed a steady decrease in desorptivity from 1.02 mm $min^{-1/2}$ for the freshly mixed mortar to 0.57 mm $min^{-1/2}$ four days after mixing. Since hydration reactions are not occurring here we suggest that the mortar batch has lost some moisture during storage.

3.4 Composition

3.4.1 Water: binder ratio

Fig. 6 shows the variation in desorptivity of CL 90 and OPC mortars at 0.5 and 0.05 MPa respectively with different water: binder ratios. It should be noted that the composition 0.78: 1 water: binder is close to that recommended for free-

flowing mortars and the mix composition 0.78: 1: 2, water: binder: sand by volume produced workable mortars for all binders. The results show an increase in desorptivity with increasing proportion of mix water, which is linear for the CL 90 mortars.

The effect of different water: binder ratio on the desorptivity of NHL 2 mortar is also shown in Fig. 2(B), in this case over a range of applied pressure. The increased proportion of water similarly causes an increase in desorptivity, with pressure having a more marked effect at the higher water: binder ratio and increasing the value of the constant C.

3.4.2 Sand: binder ratio

Fig. 7 shows the effect of the volume fraction of sieved sand on the desorptivity of freshly mixed NHL 5 paste (*i.e*. zero sand fraction) and three NHL 5 mortars of binder: sand ratios 0.5, 1 and 2. The water: binder ratio in each case was 0.78:1. Desorptivity measurements were carried out at three pressures (0.05, 0.1 and 0.15 MPa) for each mix. It can be seen that increasing the volume fraction of sand caused a decrease in desorptivity and that this result is consistent over the rage of pressures. NHL 5 paste (zero sand) was the least water retaining. The greatest increase in water retaining ability of mortars occurred between mix proportions 1:1 and 1:2 lime: sand. It can also be seen that changes in pressure became less significant as the volume fraction of sand is increased.

3.4.3 Particle size of sand

The effect of sand particle size on the desorptivity of freshly-mixed NHL 2 mortars is shown in Fig. 8. As the particle size decreases, the desorptivity is

reduced. This is because smaller particles not only provide a greater surface area for wetting but also for bonding with the binder material thereby increasing the water retaining ability.

4 Conclusions

The water retaining ability of mortars can be quantified by their desorptivity and this parameter can be measured easily and relatively quickly using a pressure cell. We recommend that measurements be made over a range of pressures in order to establish that the resultant filter cake is incompressible and are confident that this is so for mortars.

Desorptivity is dependent on the hydraulicity and particle size distribution of the binder material; hydrated lime mortars being most strongly water retaining, hydraulic lime mortars less so and Portland cement mortars much less so. Hydraulic mortars in closed containers remain water releasing for about one hour after mixing then become more water retaining as they begin to stiffen. As these effects are more pronounced for the most hydraulic binder materials we suggest they are related to the progress of hydration reactions. It would be interesting to monitor particle size distribution and pore volume changes as hydration progresses.

Water retaining ability is also strongly dependent on mortar composition. Desorptivity is increased (mortar becomes less water retaining) when water: binder ratio is increased, binder:sand ratio is increased and when the particle size of sand is increased. These findings reinforce the guidance offered on mix composition for masonry mortars (using non hydraulic lime) in BS5628 [14].

Increasing amounts of water can be desorbed from a given mortar by increasing the applied pressure, but only up to a point: a pressure limit is reached above which further increase in desorptivity does not occur. The more hydraulic the mortar the lower the pressure limit becomes. CL 90 mortars, on the other hand, are so water retaining that this pressure limit is not reached by 1 MPa. In practice, pressure is applied by the capillary potential (capillary pressure) of the masonry substrate. Typical capillary pressures exerted by clay common brick lie in the range 0.1 to 0.2 MPa [15]. These results have important practical consequences and show that, provided the brick exerts sufficient capillary pressure, *i.e.* above 0.1 MPa, minor variations in brick type are not significant in dewatering freshly mixed cement and NHL 5 mortars, but may make a difference to weakly hydraulic NHL 3.5 and NHL 2 mortars and to air lime mortars.

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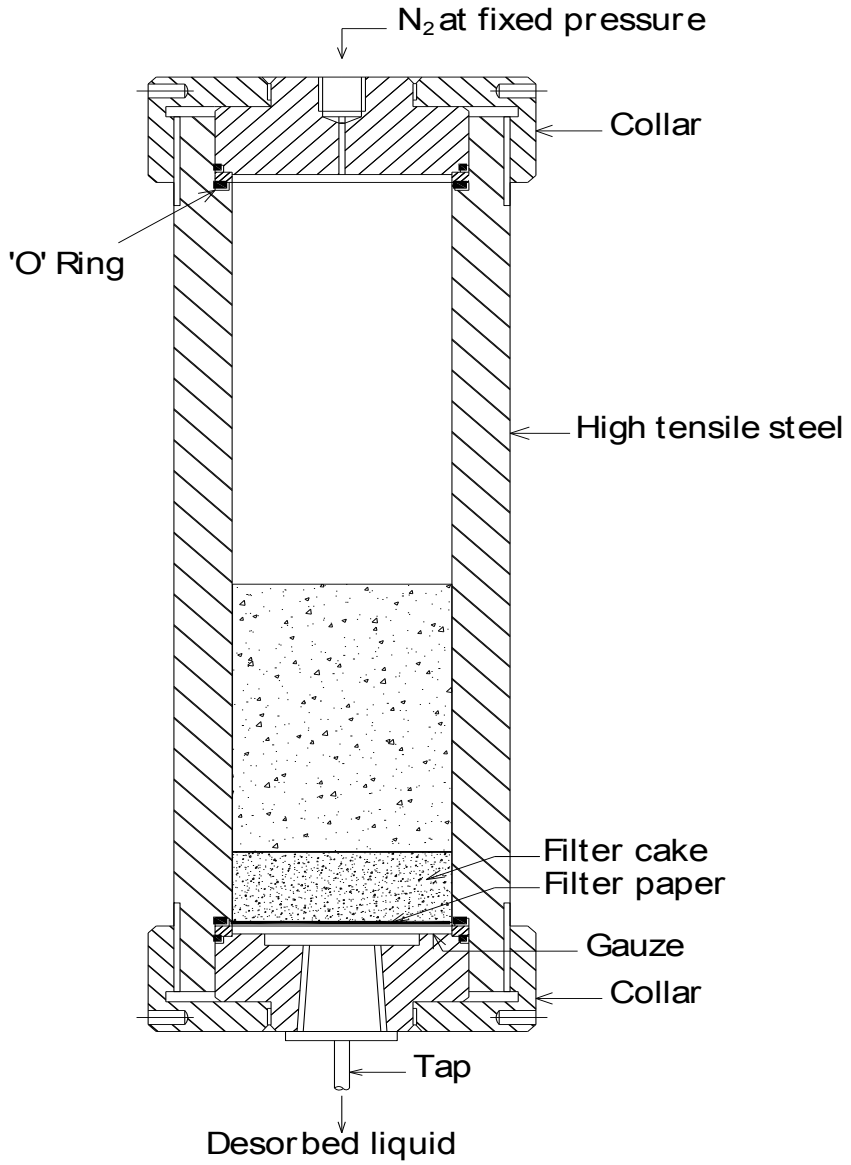

Figure 1. Schematic diagram of the pressure cell used in the measurements of desorptivity.

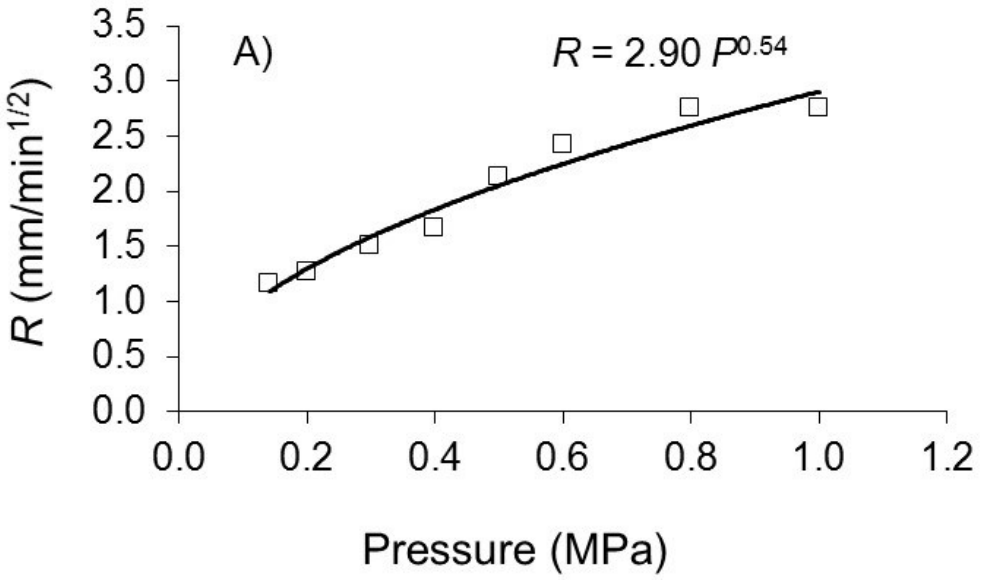

Figure 2. Variation in desorptivity with applied pressure. A: □ ,CL 90 mortar of composition 0.78:1:2 water: binder: sand; B: ●, NHL 2 mortar of composition 1:1:2 water: lime: sand (upper line) and 0.78:1:2 water: lime: sand (lower line).

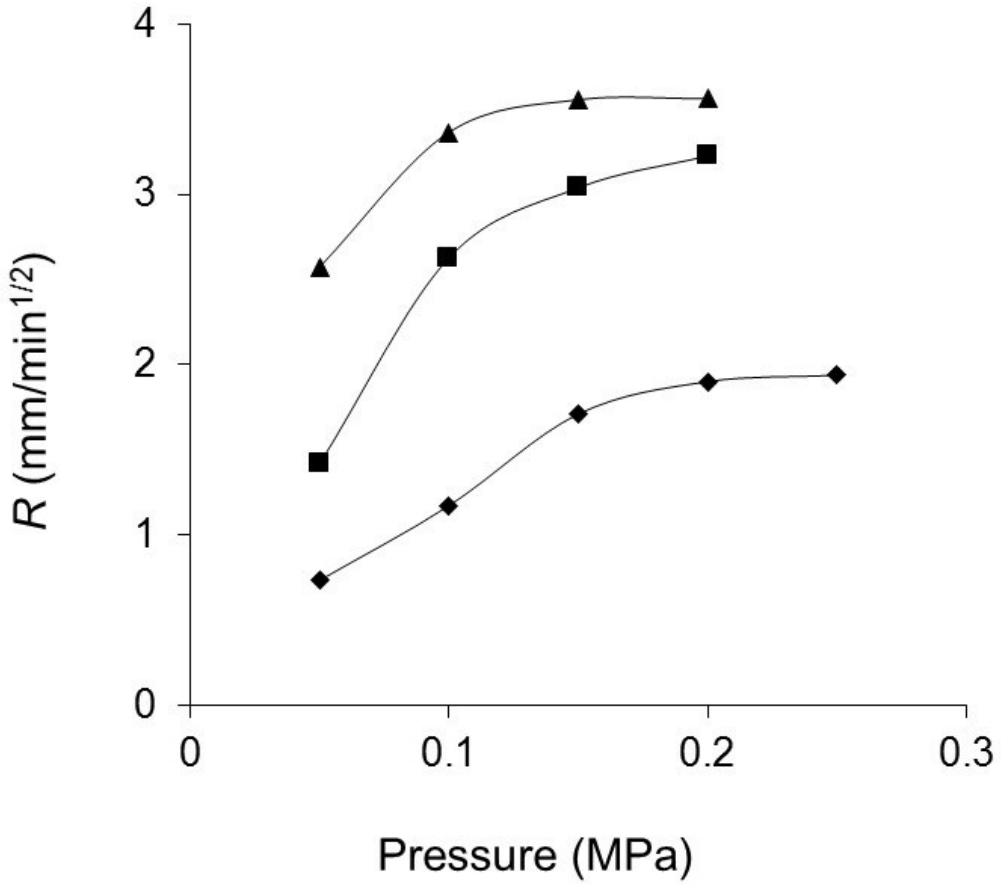

Figure 3. Variation in desorptivity with applied pressure for a range of freshly mixed mortar types all of composition 0.78:1:2 water: binder: sieved sand. ▲,OPC; ■, NHL5; ♦, NHL3.5.

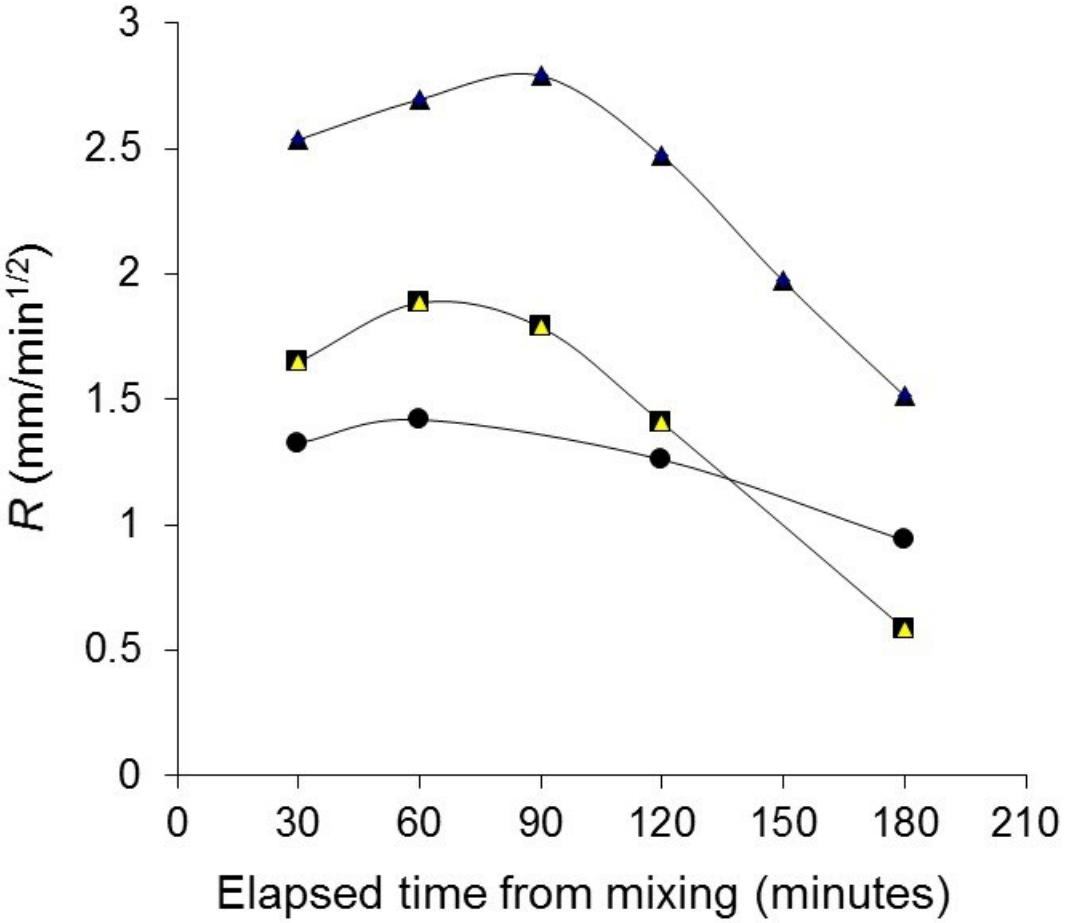

Figure 4. Change in desorptivity at 0.05 MPa applied pressure with elapsed time from mixing. ▲, OPC; ■, NHL 5; ●, NHL 2 mortars all of the same mix composition 0.78:1:2 water: lime: sieved sand.

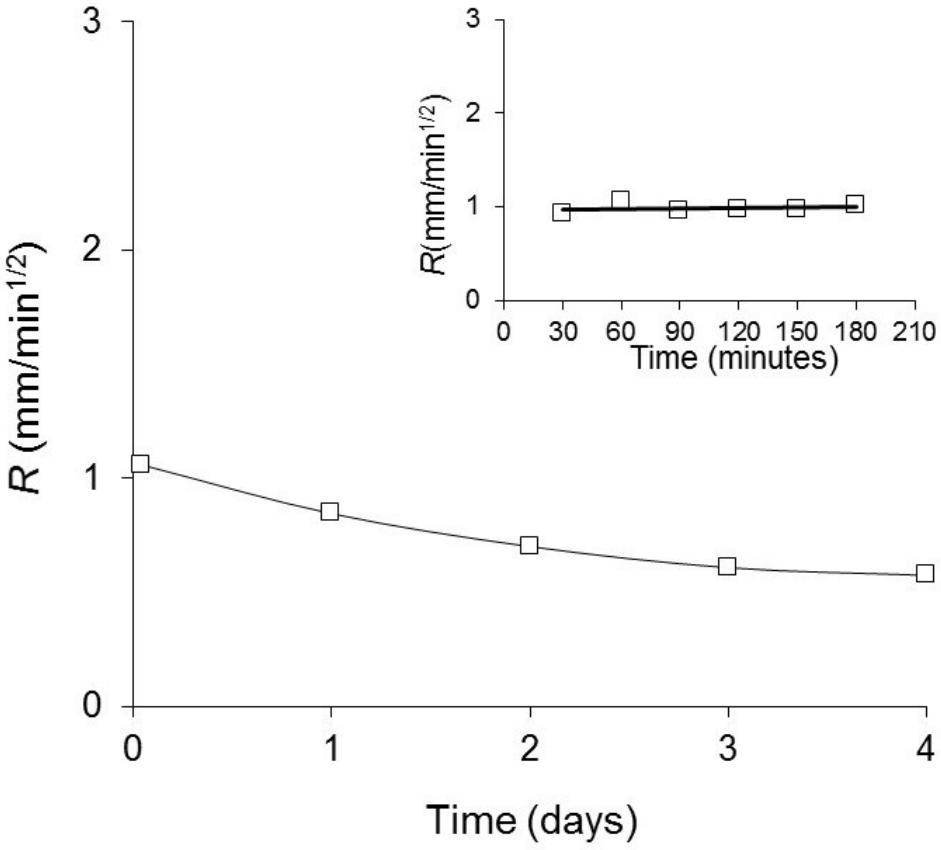

Figure 5. Change in desorptivity with elapsed time from mixing for a CL 90 mortar of mix composition 0.78:1:2 water: binder: sand over 24 hours (upper graph) and 4 days (lower graph) following mixing. All desorptivity measurements carried out at 0.2 MPa.

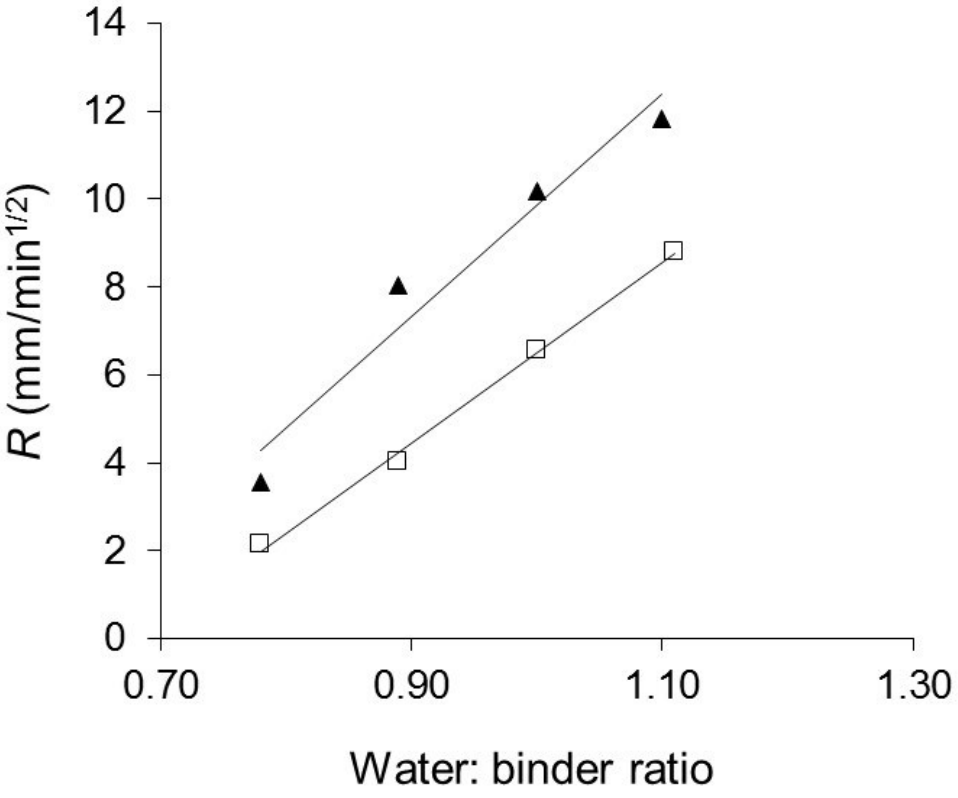

Figure 6. Variation in desorptivity with water: binder ratio for freshly mixed CL 90 and OPC mortars of composition 1:2 binder: sand. □ , CL 90 mortar at 0.5 MPa; ▲, OPC mortar at 0.05 MPa.

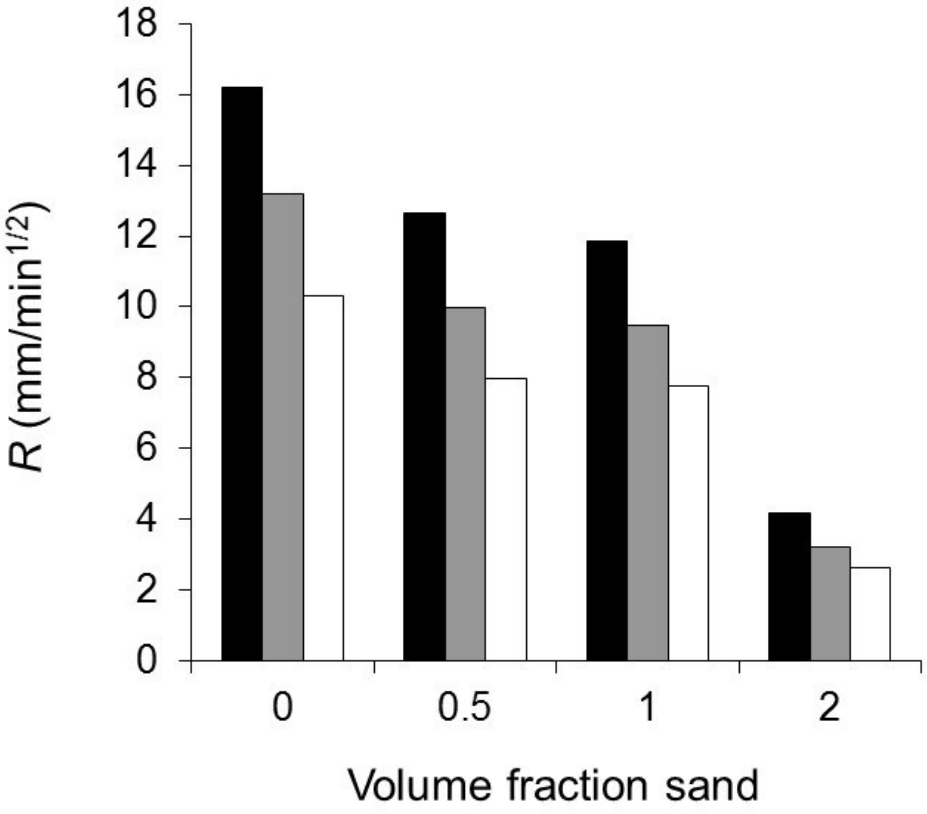

Figure 7. Variation in desorptivity with volume fraction sieved sand contents of zero, 0.5, 1 and 2 for freshly mixed NHL 5 with a constant water: binder ratio of 0.78:1 carried out at pressures of □, 0.05 MPa; ■, 0.1 MPa; ■, 0.15 MPa.

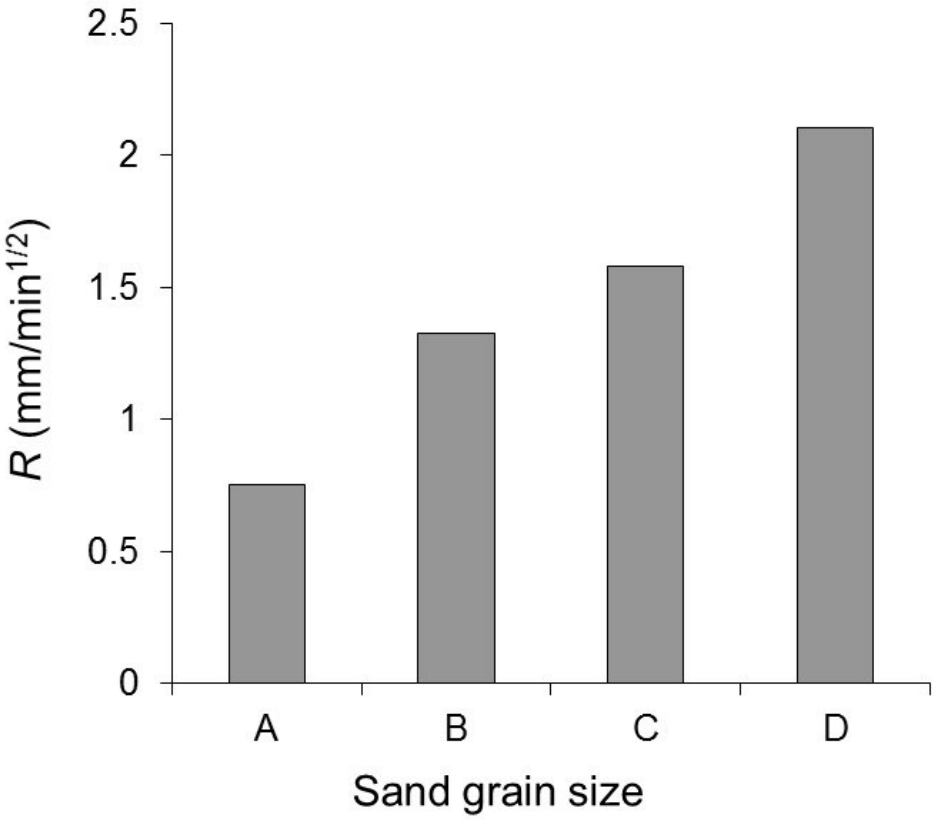

Figure 8. Desorptivity of NHL2 mortars of constant 0.78:1:2 water: binder: sand mix proportions prepared with different grain sizes of sand. A, 75 - 300 μm; B, 150 - 300μm. C, 300 - 600 μm; D, 75 - 600 μm. The applied pressure was 0.05 MPa in each case.

Tables:

	Bulk Density (g/cm^3)	Surface Area (m^2/g)
CL90	0.65	14.84
NHL2	0.71	5.80
NHL3.5	0.67	5.83
NHL5	0.89	3.12
OPC	1.12	0.95

Table 1. Bulk densities and specific surface areas of cement, hydraulic limes and air lime.

Degree of Hydraulicity	Mortar	Pressure (MPa)	Desorptivity ($mm/min^{1/2}$)
↓	OPC	0.05	2.54
	NHL5	0.05	1.65
	NHL2	0.05	1.33
	CL90	0.2	0.97

Table 2. Desorptivity values measured immediately after mixing for the range of hydraulic and non hydraulic mortars at the lowest applied pressure in each case.